DIVISION S-8—FERTILIZER TECHNOLOGY AND USE

Effect of Nitrogen Source and Dicyandiamide on Growth and Water Relations of Cotton

D. W. REEVES,* J.T. TOUCHTON, AND D. H. RICKERL

ABSTRACT

Nitrification inhibitors such as dicyandiamide (DCD) may improve N efficiency for cotton (*Gossypium hirsutum* L.) grown on sandy Coastal Plain soils. Research has demonstrated that cotton is sensitive to DCD, and field experiments suggest a possible link between cotton response to DCD and rainfall distribution. A greenhouse experiment was conducted to investigate the effect of DCD and N source on growth and water relations of cotton on a typical Coastal Plain soil. Cotton (Deltapine 90) was grown in pots containing a Norfolk sandy loam (fine-loamy, siliceous, thermic, Typic Paleudults). Nitrogen (50 mg kg^{-1}) as $NaNO_3$ or urea, and DCD (0, 2.5, 5, 10, 15, and 20 mg DCD-N kg^{-1}) were applied to the soil at first true leaf. Soil water content, leaf xylem water potential (ψ_l), and stomatal conductance were monitored during a 3-d drying period, commencing at first bloom, following which plants were harvested. Both N source and DCD affected plant growth and water relations, but there were no significant interaction effects. Fertilization with $NaNO_3$ increased leaf dry weight 9.1% compared with fertilization with urea. Plants fertilized with $NaNO_3$ depleted soil moisture faster than plants fertilized with urea, resulting in lowered stomatal conductances and more negative ψ_l throughout the drying period. Dicyandiamide lineraly reduced leaf area and dry weight, and stem dry weight. Dicyandiamide did not affect soil-water depletion, ψ_l, or stomatal conductance in the morning. Under more stressful afternoon conditions, DCD, especially at rates ≥10 mg DCD-N kg^{-1}, increased stomatal conductance over the range of available soil water. Dicyandiamide-induced increases in stomatal conductance under conditions of nonlimiting soil water could increase photosynthesis and possibly lint yield. In years when soil water is limiting, however, additional stress from DCD phytotoxicity could result in yield reductions.

Additional Index Words: ***Gossypium hirsutum* L., nitrification inhibitor, DCD, stomatal conductance, leaf water potential, NH_4^+-N, NH_3-N.**

NITRIFICATION INHIBITORS may offer an alternative to splitting N applications for improving the efficiency of N applied to cotton grown on sandy soils of the southeastern Coastal Plain. The nitrification inhibiting properties of dicyandiamide (DCD) ($H_2NC[NH]NHCN$) have been known since the early 1900s (Cowie, 1918), but only recently have formulations of N fertilizers containing DCD become commercially available. Dicyandiamide is nonvolatile, water soluble (Reider and Michaud, 1980), and chemically and physically stable (SKW Product Studies, 1983). The compound is also soluble and stable in anhydrous NH_3 (Ashworth and Rodgers, 1981). These properties enable DCD to be effectively formulated with a wide variety of N fertilizers, including urea, NH_4^+-salts, N solutions, animal manures, and anhydrous NH_3.

Dicyandiamide has been shown to increase yields of winter wheat (*Triticum aestivum* L.) (Rodgers et al., 1985; Rodgers and Ashworth, 1982) and grain sorghum [*Sorghum bicolor* (L.) Moench] (Touchton and Reeves, 1985); however, research involving DCD applications to cotton has indicated that cotton is sensitive to DCD. The only two reports of DCD applications to cotton have both described pot trials (Reddy, 1964; Reeves and Touchton, 1986). In separate experiments, Reddy (1964), applied 50 or 110 mg N kg^{-1} soil as $NaNO_3$ or $(NH_4)_2SO_4$ in combinations with 0, 3.3, 6.7, and 16.7 mg DCD-N kg^{-1} to cotton grown in a Cecil sandy loam (clayey, kaolinitic, thermic, Typic Hapludults). The 16.7 mg DCD-N kg^{-1} concentration resulted in visual phytotoxicity symptoms and reduced dry matter yields, regardless of N source. Reeves and Touchton (1986) applied 60 mg N kg^{-1} soil in varying ratios of urea/DCD to cotton grown in a Norfolk sandy loam (fine-loamy, siliceous, thermic, Typic Paleudults). Dry weights of shoots and roots were reduced as the proportion of N as DCD was increased. The effect could not entirely be accounted for by lack of availability of DCD-N as dry weights were reduced to below those of a zero-N check when >67% of the N was supplied as DCD.

Field experiments on the same soil have shown erratic responses to preplant-banded applications of urea containing DCD (unpublished data). Averaged over 3 N rates (67, 101, and 134 kg ha^{-1}), urea formulated with 10% of the N as DCD-N reduced seed cotton yield 30 and 10%, respectively, in 2 yr, but increased yield 13% in another year. Weather data indicated that in both years when yield reductions occurred, plants were subjected to drought stress prior to peak bloom. The yield increase occurred in a growing season with a more favorable rainfall distribution.

Erratic plant responses to nitrification inhibitors can be caused by a number of factors and their interactions. These include N rate applied, soil temperature, moisture, texture, pH, organic matter, and biological activity (Keeney, 1980; Slangen and Kerkhoff, 1984). The interaction of nitrification inhibitors and plant water status has not been well-researched. This interaction results from two primary causes, the inhibition of nitrification with resulting increased plant uptake of NH_4^+ ions, and the effect of the nitrification inhibitor, per se, on the plant's physiological processes.

Form of N has been shown to influence plant water relations. Ammonium-N in comparison with NO_3^--N inhibited water uptake, decreased leaf xylem water potentials (ψ_l) and increased leaf diffusive resistances of tomato (*Lycopersicon esculentum* Mill.) (Quebedeaux

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and Ozbun, 1973; Pill et al., 1978; Pill and Sparks, 1982) and Ostrich fern [*Matteuccia struthiopteris* (L.) Todaro] (Prange and Ormrod, 1982). The inhibitor nitrapyrin (2-chloro-6-[trichloromethyl] pyridine) has also been shown to reduce ψ_l and increase leaf diffusive resistance of tomato plants grown in NO_3^--N fertilized medium (Pill, 1981).

Nitrapyrin and DCD both inhibit the cytochrome oxidase involved in NH_3 oxidation by *Nitrosomonas* (Hauck, 1980), and phytotoxicity symptoms are similar for both inhibitors (Reeves and Touchton, 1986; Rufner et al., 1984). These similarities, as well as inferences from field data, suggest that DCD may affect water relations of cotton.

The objectives of this greenhouse study were to determine the effects of N form and DCD concentrations on plant growth and water relations of cotton grown in a sandy Coastal Plain soil.

MATERIALS AND METHODS

Ten seeds of the cotton cultivar 'Deltapine Acala 90' were planted in separate 22-cm-diam., 5.45-L, plastic containers containing 6.35 kg (oven-dry weight basis) of Norfolk sandy loam that had been sieved through a 2.5-mm screen. The initial soil pH was 5.8, and Mehlich I (Mehlich, 1953) P, K, Ca, and Mg (Hue and Evans, 1979) averaged 39, 76, 325, and 53 mg kg^{-1}, respectively. Organic matter content averaged 10.3 g kg^{-1} and cation exchange capacity averaged 3.6 $cmol_c$ kg^{-1}. Initial total N and inorganic N averaged 0.38 g kg^{-1} and 4 mg kg^{-1}, respectively. Ten days prior to planting, 6.0 g of dolomitic limestone (90% calcium carbonate equivalent, CCE) was mixed with the soil in each pot and each pot was watered to saturation. Pots were fertilized at planting, and weekly thereafter, with 2× Hoagland's solution (Hoagland and Arnon, 1950) minus N to ensure that no mineral deficiencies would confound results. At the first true-leaf stage of development (15 d after emergence), plants were thinned to 3 plants per pot and treatments were applied as aqueous solutions to the soil surface of each pot except the zero-N check pots. Water (0.5 L) was applied to all pots immediately after treatment applications to leach treatments into the soil.

The experimental design was a factorial arrangement of N source × DCD rates in a randomized complete block with five replications. Nitrogen sources were urea and $NaNO_3$. Nitrogen rate (apart from DCD-N) was 50 mg kg^{-1} soil. Dicyandiamide rates were 0, 2.5, 5.0, 10.0, 15.0, and 20.0 mg DCD-N kg^{-1} soil (DCD contains 67% N). A duplicate of the design was arranged on an adjacent greenhouse bench. Gypsum blocks (Soil Moisture Equipment Corp., Santa Barbara, CA),[1] calibrated for gravimetric soil-water content, were placed in each pot of one set of the experiment. Pots were watered as needed, based on gypsum block resistance readings, so that plants were not water stressed prior to the water relations measurement period. At first bloom, (45 d after treatment application, 60 d after emergence), at 1700 h CST, pots were watered to saturation (0.31 kg H_2O kg^{-1} dry soil). Stomatal conductance, leaf water potential (leaf xylem pressure potential, ψ_l), and soil-water content were measured from 0830 to 1000 and from 1400 to 1530 h CST during a 3-d drying period starting 61 d after emergence. Stomatal conductances (abaxial + adaxial conductances in parallel) of the youngest fully expanded leaves were measured with a LI-1600 steady-state porometer (LI-COR, Inc., Lincoln, NE.)[1] from the set of pots with gypsum blocks. Because of the destructive nature of ψ_l measurements, uppermost fully expanded leaves were excised from the duplicate set of pots without gypsum blocks, placed immediately in small plastic bags, and transferred to a pressure chamber (Soil Moisture Equipment Co., Santa Barbara, CA)[1] for determination of ψ_l. Technical problems prevented ψ_l measurements during the afternoon of the first day of the drying period.

Sixty-seven days after emergence, plants were harvested and separated into leaves, squares and blooms, stems, and roots. Roots were washed free of soil, blotted dry, and weighed. Leaf area was determined on a LI-COR LI-3100 area meter.[1] All plant organs were then dried for 72 h at 60 °C and weighed.

Statistical analyses included analysis of variance and regression analysis using the General Linear Models (GLM) procedure of SAS Inst. (Freund and Littell, 1981). Fisher's protected least significant difference (LSD, $P \leq 0.05$) was used to separate means among N sources. There were no N source × DCD interaction effects on any variable; therefore, only main effects are presented throughout the paper.

[1] Mention of a trademark name or a proprietary product does not constitute a guarantee or warranty of the product by the USDA or the Alabama Agric. Exp. Stn., and does not imply its approval to the exclusion of other products that may also be suitable.

RESULTS AND DISCUSSION

Plant Growth

Nitrogen Source Effects

Nitrogen applied as $NaNO_3$ increased total dry weight of cotton plants compared with fertilization with urea (Table 1). This increase was primarily the result of an increase in leaf tissue. The number of fruiting structures (squares and blooms) per plant was also increased by fertilization with $NaNO_3$ as compared with fertilization with urea. Nitrogen recovery was less ($P \leq 0.006$) for plants fertilized with urea rather than $NaNO_3$ (95.3 vs. 102.5%). Although precautions were taken to minimize urea hydrolysis and NH_3 volatilization (soil pH in zero-N check pots averaged 6.6 at the end of the experiment and all pots were watered to incorporate treatments into the soil), it is possible that NH_3 volatilization reduced the efficiency of urea.

Dicyandiamide Effects

Plant dry weight decreased linearly as DCD rate increased (Fig. 1). The decrease was due to reductions in both stem and leaf dry weights (Fig. 2). Leaf area

Table 1. Effect of N source on growth of cotton in the greenhouse 67 d after emergence.

N source	Dry wt.				Root fresh wt.	Leaf area	Squares + blooms
	Total	Roots	Stems	Leaves			
	g					cm^2	no. $plant^{-1}$
$NaNO_3$	41.63	10.56	15.19	13.76	70.30	1681.3	2.15
Urea	39.25	10.19	14.65	12.61	69.56	1592.3	1.80
LSD (0.05)	1.65	1.19 (NS)	0.71 (NS)	0.69	5.11 (NS)	92.0 (NS)	0.26
Zero-N control	10.36	3.12	3.24	4.01	23.6	432.0	0

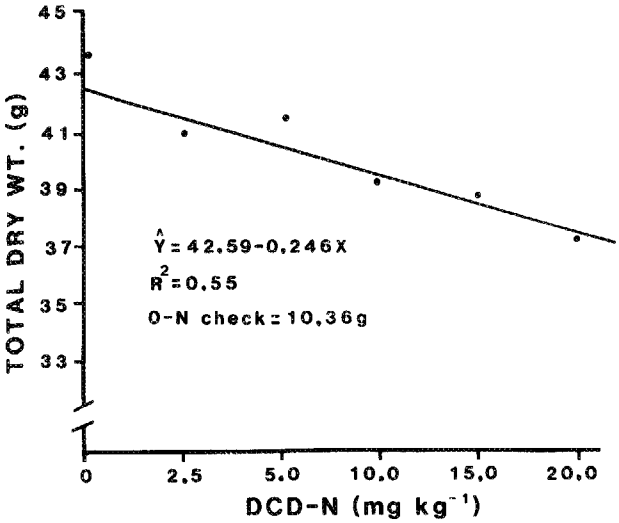

Fig. 1. Effect of DCD on total dry weight of greenhouse grown cotton harvested 67 d after emergence. The R^2 is calculated from individual data points; model significant at the 0.01 level.

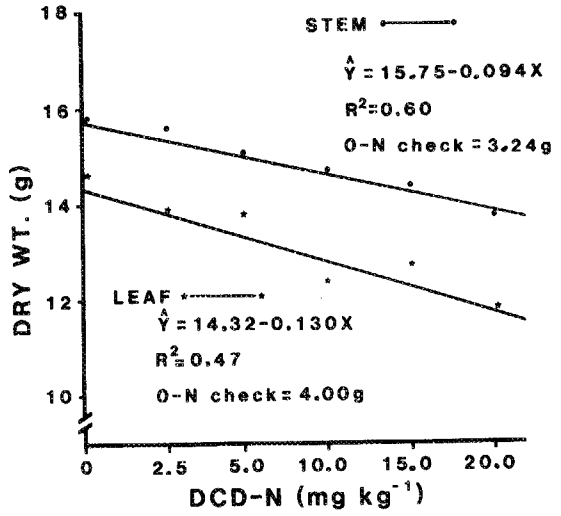

Fig. 2. Effect of DCD on dry weight of stem and leaf tissue of greenhouse grown cotton harvested 67 d after emergence. The R^2 values are calculated from individual data points; both models significant at 0.01 level.

was reduced similarly to leaf dry weight (data not shown). Dicyandiamide reduced root fresh weight (Fig. 3) but did not affect root dry weight (data not shown). Maftoun and Sheibany (1979) postulated that DCD-induced growth suppression was the result of reduced lateral root formation and main root elongation accompanied by reduced water and nutrient absorption. Amberger and Vilsmeir (1983) in pot trials with oats (*Avena sativa* L.) and spring wheat (*T. aestivum* L.) reported that water-soluble DCD was taken up by plants and located mainly in leaves and straw. They did not detect DCD in roots. Our results, here and previously (Reeves and Touchton, 1986), in conjunction with those of Amberger and Vilsmeir (1983), would suggest that the primary site of phytotoxicity of DCD lies in green tissue, and not in root tissue.

Table 2. Effect of N source on soil-water depletion and water relations of greenhouse grown cotton plants during a 3-d drying period commencing at first bloom.

N source	Days after emergence and (time of day)†					
	61 (am)	61 (pm)	62 (am)	62 (pm)	63 (am)	63 (pm)
	Gravimetric water content, kg kg^{-1}					
$NaNO_3$	0.310	0.310	0.259	0.196	0.097	0.081
Urea	0.310	0.310	0.283	0.219	0.113	0.098
LSD (0.05)	0.003 (NS)	0.003 (NS)	0.018	0.026 (NS)	0.012	0.012
Zero-N control	0.310	0.310	0.285	0.282	0.263	0.236
	Stomatal conductance, cm s^{-1}					
$NaNO_3$	1.353	0.674	1.285	0.570	0.499	0.086
Urea	1.394	0.768	1.324	0.624	0.692	0.123
LSD (0.05)	0.127 (NS)	0.070	0.148 (NS)	0.103 (NS)	0.155	0.037
Zero-N control	0.743	0.442	0.669	0.404	0.538	0.359
	Leaf water potential, −MPa					
$NaNO_3$	0.994	-‡	0.767	1.342	1.223	1.583
Urea	0.939	-	0.747	1.249	0.960	1.456
LSD (0.05)	0.073 (NS)	-	0.036 (NS)	0.085	0.183	0.121
Zero-N control	1.05	-	0.625	1.175	0.813	1.055

† Pots watered to saturation 60 d after emergence.
‡ Data not taken because of technical problems.

Plant Water Relations

Nitrogen Source Effects

The larger plants resulting from $NaNO_3$ fertilization depleted soil water faster than plants fertilized with urea (Table 2). This resulted in decreased stomatal conductances throughout the imposed drying period (Table 2). Similarly, plants fertilized with $NaNO_3$ maintained lower (more negative) ψ_l than plants fertilized with urea (Table 2). Stomatal conductances of zero-N control plants remained lower than N-fertilized plants until soil water became limiting (morning of Day 3 of drying period, 63 d after emergence). This behavior is similar to that reported for bean plants (*Phaseolus vulgaris* L.) by Shimshi (1970). He reported that transpiration rates of N-deficient plants were lower than those of N-supplied plants when soil moisture remained high, but as soil moisture approached the wilting range, transpiration rates of N-deficient plants

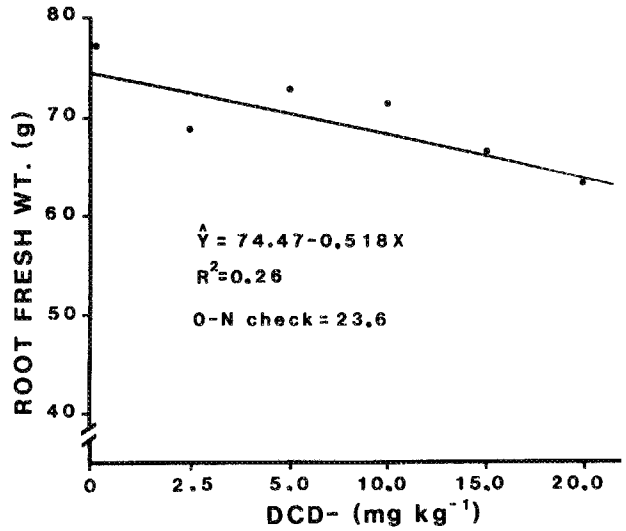

Fig. 3. Effect of DCD on root fresh weight of greenhouse grown cotton harvested 67 d after emergence. The R^2 is calculated from individual data points; model significant at the 0.01 level.

became higher than those of N-supplied plants. Research indicates that NH_4^+-N, compared with NO_3^--N, decreases ψ_l (Quebedeaux and Ozbun, 1973; Pill et al., 1978; Pill and Sparks, 1982; Prange and Ormrod, 1982). Nitrogen deficiency decreased ψ_l in greenhouse grown cotton (Radin and Parker, 1979). These findings by other researchers, as well as trends in water depletion (Table 2), indicate that differences in ψ_l among N sources or between N-fertilized plants and N-deficient plants are reflections of soil-water availability during the drying period and not the result of NH_4^+ ion uptake.

Dicyandiamide Effects

Dicyandiamide did not affect depletion of soil water or ψ_l during the dry-down period. Stomatal conductance during the morning was not affected by DCD, but under more stressful afternoon conditions, DCD affected measurements. This effect is demonstrated by regression of stomatal conductance on soil-water content (Fig. 4). Dicyandiamide, at all concentrations, increased stomatal conductance as soil water increased from 0.12 kg kg^{-1} to saturation. Soil bulk density at the conclusion of the experiment averaged 1.52 Mg m^{-3}. For this soil, at this bulk density, 0.12 kg kg^{-1} corresponds to a soil-water tension of 85 kPa. Dicyandiamide concentrations $\geq$10 mg kg^{-1} increased responsiveness of stomata to decreasing soil-water content. The effect of DCD on stomatal conductance was not a reflection of soil-water depletion, as was the lowered stomatal conductances of plants fertilized with $NaNO_3$, nor was it due to increased N uptake from mineralization of DCD-N. Nitrogen recovery data indicated no difference in N uptake among DCD rates (data not shown).

CONCLUSIONS

Both N source and DCD affected plant growth and water relations, but there were no N source × DCD interaction effects on plant growth or water relations. Decreases in stomatal conductances of $NaNO_3$-fertilized plants compared with urea-fertilized plants were a result of increased plant growth and consequent greater soil-water demand.

Dicyandiamide linearly reduced cotton growth. However, the effect was not substantial until DCD-N rate was between 5 and 10 mg kg^{-1} soil. A broadcast application of 112 kg N ha^{-1} (a normal rate for cotton on coarse-textured soils) formulated with 10% of the N as DCD-N would result in a concentration of 5 mg DCD-N kg^{-1} soil. Higher concentrations in the root zone of cotton from banded applications or from higher rates of N formulated with DCD might adversely affect cotton growth.

Dicyandiamide-induced increases in stomatal conductance over the range of available soil water may offer one explanation for erratic responses of cotton to DCD in field experiments. Nitrogen recovery data (data not shown) indicated these increases cannot be attributed to increased N uptake. Likewise, the lack of any N source × DCD interaction suggests that effects of DCD on this physiological process is the result of the compound itself and not increased plant uptake of NH_4^+-N as a result of inhibition of nitrification. The

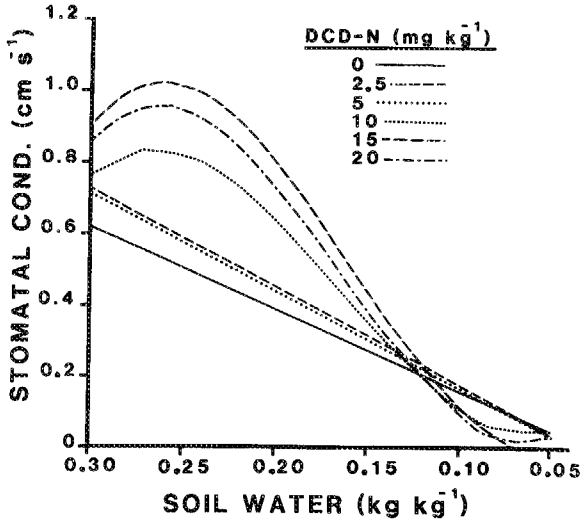

Fig. 4. Relationship between soil water and stomatal conductance of greenhouse cotton plants as influenced by DCD. The R^2 = 0.75, 0.87, 0.79, 0.81, 0.83, and 0.86 for 0, 2.5, 5, 10, 15, and 20 mg DCD-N kg^{-1}, respectively (calculated over individual data points). All models are significant at the 0.05 level or greater.

DCD-induced increases in stomatal conductance under conditions of nonlimiting soil water could increase photosynthesis and possibly lint yield. In years when soil water is limiting, however, additional stress from DCD phytotoxicity could result in yield reductions.

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DIVISION S-9—SOIL MINERALOGY

Identification of Clay Minerals in Soil: The Effect of Sodium-Pyrophosphate

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ABSTRACT

The effect of $Na_4P_2O_7$ on soil clay minerals, layer silicate clays, and chloritized montmorillonite (synthetic) was investigated by x-ray diffraction. The soil samples were treated with 30% H_2O_2, boiled, and water-washed by centrifugation. The resulting organic-free solids were dispersed as follows: shaken with 0.1 *M* $Na_4P_2O_7 \cdot 10H_2O$ for 16 h only; treated with dithionite–citrate–bicarbonate (DCB); and treated with DCB followed by 0.1 *M* $Na_4P_2O_7 \cdot 10H_2O$. The 0.1 *M* $Na_4P_2O_7 \cdot 10H_2O$ treatment caused the peak at 1.403 nm in the soil samples to become enlarged, sharp, and move to 1.339 nm. Other sharp peaks were also produced at 0.666, 0.637, 0.590, 0.561, 0.444, and 0.334 nm. These peaks were not observed in the x-ray diffractograms of kaolinite and halloysite, but those of montmorillonite, chloritized montmorillonite, and chlorite were very similar to those of the soils. Heat treatment at 100°C destroyed the 0.637-, 0.590-, and 0.561-nm peaks in some of the soil clay samples. At 300° the remaining peaks disappeared suggesting that the sharp peaks resulting from the 0.1 *M* $Na_4P_2O_7$ treatment may be the result of mineral hydration. Some of these new peaks from 0.1 *M* $Na_4P_2O_7$ treatment of soil clays, the 0.334-nm peak for example, can be mistaken as indicative of quartz in samples that do not contain this mineral. Thus the continued use of this reagent for mineralogical studies is not recommended.

Additional Index Words: **hydrogen peroxide, soil dispersion, sodium hexametaphosphate, sodium pyrophosphate.**

PRETREATMENT OF SOIL with hydrogen peroxide (H_2O_2), followed by the addition of sodium hexametaphosphate [$(NaPO_3)_6$] for dispersion, alters some soil minerals (Drosdoff and Miles, 1938; Bourget and Tanner, 1953; Martin, 1954; Jackson, 1956) but this procedure is still used in many tropical laboratories for mineralogical studies. In some laboratories, $(NaPO_3)_6$ has been replaced by Na–pyrophosphate ($Na_4P_2O_7$) because Calgon® [former trade name for $(NaPO_3)_2$] was no longer suitable as a dispersing agent (Yaalon, 1976). The use of $(NaPO_3)_6$ and $Na_4P_2O_7$ has persisted in the tropics because most of the soil clays are largely kaolinitic, for which the detrimental effects of $(NaPO_3)_6$ are considered negligible.

In the course of our mineralogical studies of some Nigerian soil clays, which are predominantly kaolinitic, we employed Na–hypochlorite (NaOCl) and $Na_4P_2O_7$ separately to cause dispersion. Upon x-ray diffraction (XRD), the unique features of clays dispersed with $Na_4P_2O_7$ prompted this investigation of the effect of $Na_4P_2O_7$ on silicate clay minerals. The main objectives of the work reported here are (i) to show that dispersion of soil with $Na_4P_2O_7$ results in some reaction with silicate clay minerals in the soil, and (ii) to identify the soil mineral(s) that are involved in the reaction.

MATERIALS AND METHODS

Twenty-one soil samples from three soil profiles in Nigeria were collected. Chemical and physical characteristics of these soils, which are all Ultisols, are reported in Table 1. Faceville B22t from the USA was included as an example of an Ultisol from the temperate region.

For the pretreatment with H_2O_2, 25 g of soil was weighed into a 250-mL centrifuge plastic bottle, followed by 150 mL of distilled H_2O, and 25 mL of 30% H_2O_2. Samples prepared in this manner were heated in a water bath at about 70 °C for 6 h, then cooled. All samples were water-washed twice by centrifugation. These organic-free soils were then dispersed using the following three different procedures:

(i) The sample was shaken with $Na_4P_2O_7$ for 16 h only. In brief, the 0.1 *M* $Na_4P_2O_7 \cdot 10H_2O$, which was prepared by dissolving 50 g of the salt in 1 L of distilled H_2O, was employed. To each of the 25-g samples of organic-free soil, 150

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